PATENT COOPERATION TREATY

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 10 April 2001 (10.04.01)	
International application No. PCT/GB00/02785	**Applicant's or agent's file reference** CRO 50828/WO
International filing date (day/month/year) 24 July 2000 (24.07.00)	**Priority date** (day/month/year) 10 August 1999 (10.08.99)
Applicant WOODHOUSE, Mark, Antony et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

23 February 2001 (23.02.01)

[ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was

[ ] was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland**	Authorized officer Olivia TEFY
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	**FOR FURTHER ACTION**	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
CRO 50828/WO		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/GB 00/ 02785	24/07/2000	10/08/1999
Applicant		
CROSFIELD LIMITED et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

a. With regard to the **language,** the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

[ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

[ ] contained in the international application in written form.

[ ] filed together with the international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readble form.

[ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**

[X] the text is approved as submitted by the applicant.

[ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract,**

[X] the text is approved as submitted by the applicant.

[ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. ---

[ ] as suggested by the applicant.

[ ] because the applicant failed to suggest a figure.

[ ] because this figure better characterizes the invention.

[ ] None of the figures.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
CRO 50828/WO		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB00/02785	24/07/2000	10/08/1999
International Patent Classification (IPC) or national classification and IPC C11D17/00		
Applicant INEOS SILICAS LIMITED		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 5 sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☒ Certain documents cited
   - VII ☒ Certain defects in the international application
   - VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
23/02/2001	24.10.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Giese, H-H Telephone No. +49 89 2399 8488

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/02785

I. Basis of the rep rt

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:
Description, pages:

1-12 as originally filed

Claims, No.:

1-22 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-22
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-22
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-22
	No:	Claims	

2. Citations and explanations
see separate sheet

VI. Certain documents cited

1. Certain published documents (Rule 70.10)

and / or

2. Non-written disclosures (Rule 70.9)

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

R It m V

1. **Cited documents**

The following documents (D) are referred to in this communication:

D1: WO-A-98/55583
D2: US-A-5 916 866
D3: US-A-4 414 130

2. **Novelty** (Article 33(2) PCT)

The present independent product claim 1 defines cleaning composition having granules. The granules comprise a water insoluble inorganic material and no more than 20% of water-swellable agent. The granules are combined with active ingredients in a compact moulded body. The present independent product claim 20 defines a granular composition having a water insoluble inorganic material and no more than 20% of a water-swellable agent. The present independent process claim 21 defines the preparation of a cleaning composition comprising (1) forming a granular disintegrant composition according to preceding claims and (2) mixing said granular composition with at least one more detergent-active compound.

As compared to the present applications documents D1 (see page 1, §3; page 2, §2, 3; example 1, 2; claims) and D2 (see col. 2, l. 1-17; col. 5, l. 63 to col. 5, l. 2; example 1, 2) lack in a co-granule consisting of inorganic material and water-swellable agent, and D3 (see col. 1, l. 9-13; col. 2, l. 64 to col. 3, l. 10; col. 18, l. 19-21; claim 1) lacks in a compacted moulded body.
Therefore, claims 1 to 22 are novel (Article 33(2) PCT).

3. **Inventive Step** (Article 33(3) PCT)

The problem to be solved was to provide a particulate cleaning composition which is sufficiently robust but readily breaks up and dissolves in the aqueous medium. Furthermore the cleaning granules do not result in undue deposition on the substrate being cleaned.
The solution proposed by the present application is the co-granulation of water-swellable disintegrating agent with an insoluble or sparingly soluble, hydrophilic solid before incorporated into a tablet (see application page 2, l. 1-10).

D1 deals with the same problem but solves it in a different way (see e.g. claim 14 of D1).
The cleaning composition of the present application appears to be an alternative solution to the problem which is not taught and which cannot be deduced from the prior art documents in a reasonable way.

Therefore the present application is considered involving an inventive step (Article 33(3) PCT).

Re Item VI

Certain published documents (Rule 70.10)

Application No Patent No	Publication date *(day/month/year)*	Filing date *(day/month/year)*	Priority date *(valid claim)* *(day/month/year)*
DE-A-199 01 063	20.07.2000	14.01.1999	-

Document DE-A-199 01 063, although not constituting prior art within the meaning of Rule 64(1)(b), appears to disclose all the features of present claims (see page 3, l. 56 to page 4, l. 6; page 4, l. 26-30; claims 1, 9).

Re Item VII

As required by Rule 5.1(a)(ii) PCT, it is appropriate to identify documents D1 to D3 and to discuss the relevant background art disclosed therein briefly (see also the Guidelines II, 4.4 PCT).

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

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15 February 2001 (15.02.2001)

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(71) Applicant *(for all designated States except US)*: **CROSFIELD LIMITED** [GB/GB]; Bank Quay, Warrington, Cheshire WA5 1AB (GB).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **WOODHOUSE, Mark, Antony** [GB/GB]; 67 Dundee Close, Fearnhead, Warrington, Cheshire WA2 oUJ (GB). **FREANEY, Michael, Damian** [GB/GB]; 41 Moore Avenue, Thelwall, Warrington, Cheshire WA4 2JN (GB). **KREISCHER, Pieter, Hubertus** [NL/NL]; Karposthegge 35, 6225 KJ Maastricht (NL).

(74) Agents: **JACKSON, John, Derek**; Crosfield Limited, Bank Quay, Warrington WA5 1AB et al. (GB).

(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

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Published:
— *With international search report.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/11000 A1

(54) Title: CLEANING COMPOSITIONS

(57) Abstract: A cleaning composition formed into moulded bodies for use in for example fabric washing, dishware washing, stain removal and water softening incorporates disintegrant granules comprising a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20 % of the combined weight of said inorganic material.

CLEANING COMPOSITIONS

This invention relates to cleaning compositions and, in particular, particulate cleaning compositions to be formed into moulded bodies for use in, for example, fabric washing, dishware washing, stain removal and water softening.

Detergent compositions in the form of tablets are widely described and are currently enjoying increasing popularity with consumers. They are described, for example, in GB 0 911 204 (Unilever), US 3 953 350 (Kao), DE 19 637 606 (Henkel), EP 0 711 827 (Unilever) and WO 98/40463 (Henkel). Tablets for machine dishwashing applications are described in, for example, WO 96/28530 (P&G). Tabletted detergents and cleaning compositions have several advantages over powdered or liquid compositions: they are easier to dispense and handle, do not require measuring to obtain the correct dose and, being compact, are more economical to store and transport.

Tablets of cleaning compositions are generally made by compression or compaction of a quantity of the composition in the form of particles. Production of tablets which are sufficiently hard and strong to withstand storage and handling requires a relatively high pressure to be used in this compaction process. It is then necessary that, despite this compaction, the tablets are able to disperse and dissolve rapidly when added to wash water.

One approach to achieving good dispersion of the tablet is to include in the tablet a particulate insoluble but water-swellable agent. These particles then swell with ingress of water, leading to stresses in the tablet and thence to break-up of the tablet. Thus WO 98/55583 (Unilever) describes the use of 'water-insoluble, water-swellable polymeric material' which 'promotes disintegration of the tablets in water'. Typical swelling agents which have been disclosed as possible tablet disintegrating agents are starches, cellulose and cellulose derivatives, alginates, dextrans, cross-linked polyvinyl pyrrolidones, gelatines and formaldehyde casein as well as a wide variety of clay minerals and certain ion-exchange resins.

These water swelling agents have no function in fabric washing except to aid tablet disintegration. Furthermore, because they are insoluble and of relatively large particle size, they tend to deposit on clothes during the wash (see, for example, WO 98/55575 (Henkel) and WO 98/55582). As a result, several attempts have been made to minimise the deposition of these disintegrants, for example by combining such a water-swellable, insoluble disintegrant with a second, highly soluble disintegrating aid – see WO 98/55582 (Unilever). Other attempts have included use of a preferred particle size of the disintegrant. Thus, for example, WO 98/55583 (Unilever) claims use of such material at a particle dimension of at least 400 μm to give more efficient disintegration. On the other hand, WO 98/55575 (Henkel) teaches the use of cellulose disintegrating aids with a particle size of less than 100 μm, in order to minimise deposition. This material is co-granulated with 'microcrystalline cellulose and/or one or several ingredients of detergents and cleaning agents'.

Surprisingly, we have now found that if a water-swellable disintegrating aid is co-granulated with an insoluble or sparingly soluble, hydrophilic solid before being incorporated into a tablet, then the disintegrant is much more efficient in disintegrating the tablet in use. Thus, less of the disintegrating aid needs to be added for effective disintegration, lowering the probability of deposition of the disintegrating aid on the substrate being cleaned.

The present invention seeks to provide a particulate cleaning composition in the form of a moulded body and which is relatively inexpensive to manufacture and sufficiently robust to withstand handling during production and packaging processes but readily breaks up and dissolves when contacted with an aqueous medium during the cleaning process for which it is intended without resulting in undue deposition on the substrate being cleaned.

According to the present invention there is provided a cleaning composition, the composition including disintegrant in the form of granules comprising a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular disintegrant, the granules being combined with the active ingredients of the cleaning composition in a compacted moulded body.

According to a particular aspect of the invention, a granular composition suitable for use in a cleaning composition consists essentially of a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular composition.

By "water insoluble", we mean a compound with a water solubility of less than 5 grams, preferably less than 1 gram, per 100 grams water (at a temperature of 25°C).

Preferably, the water-swellable agent comprises, in its anhydrous state, no more than 15%, more preferably no more than 10%, of the combined weight of said inorganic material and said agent of the granular disintegrant. In a typical composition of the invention, the water-swellable agent comprises, in its anhydrous state, no more than 8%, e.g. 7.5% or less, of the combined weight of said inorganic material and said agent. Generally, at least 1% of the combined weight of the water-swellable agent and the inorganic material in the granules comprises water-swellable agent.

A feature of the invention is the relatively small amount of water-swellable agent that may be employed while securing satisfactory properties for the compacted cleaning composition. Frequently, the amount of water-swellable agent in the cleaning composition is less than 2% by weight. Preferably, the amount is less than 1% of the cleaning composition but, usually, at least 0.2% of the water-swellable agent is present in the cleaning composition.

In one embodiment of the invention the inorganic material used in the formation of the disintegrant granules comprises a silica.

In a second embodiment of the invention the inorganic material used in the formation of the disintegrant granules conveniently comprises a material which acts as a functional ingredient of the cleaning composition. In this instance, the inorganic material may be an aluminosilicate such as zeolite P, A or X or mixtures thereof, with zeolite P being preferred.

Alkali metal aluminosilicates, especially zeolites, are commonly used in detergent compositions as a detergency builder. Where a cleaning composition of the invention is one containing an alkali metal aluminosilicate as detergency builder, preferably at least part of the aluminosilicate constituent of the cleaning composition is employed as the inorganic material used in the formation of the disintegrant granules. For instance, the aluminosilicate constituent typically comprises about 10 to about 60% by weight of the cleaning composition and preferably, when zeolite is used in the disintegrant granules and as a detergency builder, then at least 1% of the detergent composition comprises a zeolite constituent employed in the form of disintegrant granules.

The water-swellable agent preferably has an average primary particle size up to about 600 μm, but, conveniently, has an average primary particle size of no more than 200 μm, preferably no more than 100 μm, and a water swelling capacity of at least 5 ml/gram, preferably 10 ml/gram and more preferably 20 ml/gram as determined in the test described hereinafter.

Typically the water-swellable agent comprises polymer, frequently a wholly or partially cross-linked polymer, e.g. natural cellulose, cross-linked cellulose, (sodium) carboxy-methyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross linked starch, or cross linked polyvinyl pyrrolidone. Currently preferred are Aquasorb A500 (ex Hercules) and Ac-Di-Sol (ex FMC Corp).

The moulded body formed using the cleaning composition of the present invention may consist wholly of the cleaning composition or alternatively the moulded body may comprise a number of discrete portions, at least one of which comprises a cleaning composition in accordance with the invention. In this event, the remaining portion or portions of the moulded body may be constituted by at least one other ingredient, usually one suitable for use in detergent and cleaning applications such as fabric washing, dishware washing, stain removal and water softening.

Cleaning compositions of the invention may also contain, as essential ingredients, one or more detergency builders (wholly or partly incorporated in the disintegrant granules), and/or one or more detergent-active compounds which may be chosen from soap and non-soap anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof and/or other conventional additives.

A further aspect of the invention comprises a process for the preparation of a cleaning composition comprising forming a granular disintegrant composition consisting essentially of a water insoluble inorganic material and a water-swellable agent which, in its

anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular composition and mixing said granular composition with one or more detergent-active compounds and, optionally, with one or more detergency builders.

Alkali metal aluminosilicates are favoured as environmentally acceptable water-insoluble builders, e.g. zeolites A, X and P or mixtures thereof. Other inorganic detergency builders include layered sodium silicate as described in US-A-4 664 839 and marketed by Hoechst as SKS-6, and alkali metal (generally sodium) carbonate. Water-soluble phosphorous containing detergency building compounds such as alkali metal orthophosphates, metaphosphates, pyrophosphates and polyphosphates may be used. Also possible are organic detergency builders such as polycarboxylate polymers, e.g. polyacrylates, acrylic/maleic polymers, and acrylic phosphonates, monomeric polycarboxylates, e.g., gluconates, oxydisuccinates, glycerol mono- di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates and hydroxyethyliminodiacetates.

Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.

The preferred detergent-active compounds that can be used are soaps and synthetic non-soap anionic and nonionic compounds. Anionic surfactants are well known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly sodium linear alkylbenzene sulphonates having an alkyl chain length of $C_8 - C_{15}$; primary and secondary alkyl sulphates, particularly sodium $C_8 - C_{15}$ primary alcohol sulphates; olefin sulphonates; alkane sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, and ethoxylates of esterified fatty acids, especially the $C_9 - C_{15}$ primary and secondary alcohols ethoxylated with an average of from 3 to 20 moles of ethylene oxide per mole of alcohol.

The choice of surfactant, and the amount present, will depend on the intended use of the detergent composition. For example, for machine dishwashing a relatively low level of a low-foaming nonionic surfactant is generally preferred. In fabric washing compositions, different surfactant systems may be chosen, as is well known by the skilled detergent formulator, for handwashing products and for machine washing products.

The total amount of surfactant present will of course depend on the intended end use and may be as low as 0.5% by weight of the total composition, for example, in a machine dishwashing composition, or as high as 60% by weight of the total composition, for example, in a composition for washing fabrics by hand. For fabric washing compositions in general, an amount of from 5 to 40% by weight of the total composition is generally appropriate.

Generally, cleaning compositions of the invention will contain from 1 to 20% by weight of the disintegrant granules based on total weight of the composition. Frequently the cleaning compositions will contain from 4 to 10 % by weight of the disintegrant granules.

A suitable type of cleaning composition suitable for use in most automatic fabric washing machines contains both anionic and nonionic surfactants. Cleaning compositions according to the invention may also suitably contain a bleach system. Machine dishwashing compositions may suitably contain a chlorine bleach, while fabric washing compositions may contain peroxy bleach compounds, for example, inorganic persalts or organic peroxyacids, which may be employed in conjunction with activators to improve bleaching action at low wash temperatures. Again, the skilled detergent worker will have no difficulty in applying the normal principles to choose a suitable bleach system.

Other materials that may be present in detergent compositions of the invention include sodium silicate, fluorescers, antiredeposition agents, inorganic salts such as sodium sulphate, enzymes, lather control agents or lather boosters as appropriate, pigments, and perfumes. This list is not intended to be exhaustive.

Procedures and Tests

Granule Production

The granules of inorganic material and swelling agent may be prepared by any of the methods that will be known to those skilled in the art, e.g. by blending the dry ingredients in a mixer (such as a Pek mixer available from George Tweedy & Co of Preston - 28lb S.A. Machine) and compacting on a roller compactor (Alexanderwerk WP50 - manufactured by Alexanderwerk AG, D 5630 Remscheid 1, Germany). A typical preparative method is now described in detail with reference to silica as the inorganic material. Silica and water swelling organic particulate are blended together, in the appropriate proportions, in a Pek mixer for 30 minutes. A minimum of 2 kg of blended material so prepared is compacted by feeding into an Alexanderwerk roller compactor, fitted with a sintered block vacuum deaeration system. The roller pressure setting is selected according to the strength of granule desired, higher pressures leading to stronger granules. Generally, roller pressure is between 8 and 25 MPa and a typical roller pressure is 10 MPa. The compacted material from the compactor is fed into a granulator, which forms part of the machine, and forced through a mesh and the resulting granules are then screened to the desired particle size range, e.g. an average particle size of 250 to 1500 μm, using standard laboratory sieves. Preferably, the particles have a size range of 500 to 1200 μm.

Tablet Production

Tablets used in the Examples that follow were produced using a 45 mm diameter die set (stainless steel) in conjunction with a Universal Testing Machine Type No. Z030 from Zwick GmbH, Ulm, Germany. A known quantity, 40-45 grams, of the cleaning composition which

comprised the compositions mentioned in the Examples below was placed in the die, the die plunger was inserted and the assembly was placed between the platens of the Zwick machine which was operated to apply a predetermined pressure to produce a tablet having a defined density and, in particular, a dimensionally stable and fracture resistant tablet. Tablets thus produced were cylindrical in shape, with a diameter of 45 mm and a height of about 20 mm. Tablets with different densities were produced in order to determine the tablet disintegration and conductivity profiles. Tablets prepared had densities in the range 1250-1450 kg/m^3, which are typical values for commercial fabric washing tablets found in the Western European market in 1998-9.

Determination of tablet disintegration profile

Method 1 (Dynamic)

The tablet disintegration profile provides an indication of the extent to which various tablets (e.g. different compositions, different densities) disintegrate under the defined conditions.

4500 g of demineralised water at 20°C were added to a 5 litre vessel fitted with pH, conductivity and temperature probes and maintained at a constant temperature of 20°C by immersion in a water bath. The tablets to be tested were inserted into a metal cage having the dimensions 9 cm x 4.7 cm x 2.7 cm and having 16 apertures (each about 2 mm square) per cm^2. The metal cage was attached to the shaft of an overhead stirrer (Heidolph/Janke and Kunkel stirrer) to allow it and its contents to be rotated while immersed in the demineralised water. Prior to testing, the empty cage was immersed in the demineralised water and rotated at 80 rpm for a short period of time until the temperature of the demineralised water as detected by the temperature probe had stabilised at 20 ± 0.2°C. At this time, the conductivity, pH and temperature values registered by the respective probes were recorded. The stirrer was then switched off to allow the cage to be raised out of the water so that a pre-weighed detergent tablet to be investigated could be inserted into the cage. The cage was then re-immersed in the demineralised water together with the inserted tablet and the stirrer was switched on to resume rotation of the container at 80 rpm. Measurements of conductivity and pH, initially at 15 second intervals for one minute and thereafter at one minute intervals, were made over a period of 10 minutes after which time the cage was raised out of the demineralised water to allow the residue of the tablet to be removed. The residue was then dried in an oven at 105°C so that the dry weight of the residue could be calculated as a percentage of the original tablet weight. This procedure was repeated for a number of tablets having different compositions and different densities.

Method 2 (Static)

4500g of tap water at 20° C were added to a 5 litre vessel, which was maintained at 20° C by immersion in a water bath. The tablets to be tested were weighed and inserted into a

metal cage 20 cm in diameter with 1 cm^2 perforations. The cage was lowered into the 5 litre vessel and left for 60 seconds. The cage was then removed from the water, residue of the tablets was placed on an aluminium tray, dried for 24 hrs at 105° C and weighed to determine the percentage which had not disintegrated.

Determination of Tablet Conductivity Profile

The tablet conductivity profile provides an indication of the extent to which various tablets (e.g. different compositions, different densities) dissolve under the defined conditions. Conductivity measurements were obtained from the conductivity probe mentioned in Method 1 above at the same time as the tablet disintegration was measured.

Measurements of conductivity and pH were taken, initially at 30 second intervals for ten minutes and thereafter at one minute intervals for a further 20 minutes or until the conductivity measurements were observed to have reached a plateau corresponding to substantially total dissolution of the soluble portion of the tablet, i.e. equilibration of the tablet with water. This procedure was repeated for a number of tablets having different compositions and different densities.

Determination of water swelling capacity of water-swellable agent

To demonstrate the water swelling capacity of the water-swellable agent, 19.6 g of the agent was blended with 0.4 g of ultramarine pigment and compressed into a tablet using a laboratory tablet press at about 250 Mpa to give a tablet 32 mm in diameter. This was crushed and sieved to give granules 500-1000 µm in size. A glass tube, 33 mm in internal diameter and about 30 cm long with a sintered porous glass disc (porosity 1) fitted at one end was immersed upright, with said one end lowermost, in a large beaker of water (at 25° C) so that the water level rose to about 14 cm above the sintered glass. 1 g of granules was added to the tube and allowed to settle onto the sintered glass disc. With this arrangement water has access to the granules from both above and below. The granules immediately began to swell, forming a jelly-like mass. The ultramarine pigment imparted a blue colour to the mass making it easy to see the top and to record its height. The height of the swelling mass was recorded at intervals and showed an initial rapid rise followed by a levelling off after about 20-30 minutes. From the diameter of the tube, the volume of the swollen mass can be calculated. The result was expressed as ml/g water-swellable agent after 20 minutes.

EXAMPLE 1

Conductivity and disintegration profiles were investigated for a number of tablet formulations and densities, all based on concentrated Persil (Registered Trade Mark) original non-Biological detergent powder as manufactured by Lever Brothers of Kingston–upon-Thames, UK - formulation as sold in the UK. The detergent powder was blended with different amounts of disintegrant granules. In each instance, the disintegrant granules comprised a water

insoluble inorganic material, specifically a zeolite or a silica or a combination thereof, and a water-swellable agent. For comparative purposes, granules consisting of Persil powder alone or comprising a water-swellable agent or a mixture of a water soluble material, specifically sodium silicate or sodium carbonate, and a water-swellable material were also prepared and tested. In the Examples given below, Doucil A24 (Trade Mark) is a P-type zeolite sold by Crosfield Limited of Warrington, UK; SD2255 is a silica also obtainable from Crosfield Limited; Doucil 4A (Trade Mark) is a 4A zeolite obtainable from Crosfield Limited; the sodium disilicate is obtainable from Crosfield Limited under the trade mark Pyramid 95; the sodium carbonate is obtainable from Solvay Chemicals Limited (identified in their product literature as Soda Ash (Sodium Carbonate) Light Rheinberg); and the water-swellable agent was sodium carboxymethylcellulose as sold under the trade mark Aquasorb A500 by Hercules Limited of Salford, UK.

TABLE 1

Property	SD2255	Doucil A24	Doucil 4A	Sodium Disilicate	Soda Ash	Aquasorb
Surface Area (m^2/g)	650	NM	NM	NM	NM	NM
Pore Volume (ml/g)	1.3	NM	NM	NM	NM	NM
APS (μm)	5	1.2*	3*	100*	See below	40*
Moisture Content (% by weight)	2	10*	20*	18-20*	< 1.5	5*
Oil absorption (g/100g)	228	60*	40*	NM	NM	NM

NM = Not measured

In Table 1, APS represents average particle size (d_{50}) as measured using a Malvern Mastersizer (Trade Mark) obtainable from Malvern Instruments in the UK and the values marked * are taken from typical data for the product or from specifications supplied by the manufacturer. The Soda Ash used in the Examples had a bulk density of 0.53 kg/dm^3 and was found to have a particle size distribution (by sieve analysis) as follows:

> 1000 μm = 1wt %

500 - 1000 μm = 0.5 wt %

250 - 500 μm = 3.5 wt %

75 - 250 μm = 74.5wt %

0 - 75 μm = 20.5wt %

The different formulations of the disintegrant granules (on a by weight basis) are given in Table 2.

TABLE 2

Granule Code	Wt. % of Aquasorb A500 in granule	Remainder of Granule (Wt. % and Material)
A	10	90% SD2255 Silica
B	10	45% SD2255 Silica, 45% Doucil A24
C	10	90% Doucil 4A
D	10	90% Doucil A24
E	7.5	92.5% Doucil A24
F	5	95% Doucil A24
G	10	Pyramid 95
H	10	Sodium Carbonate

The granules coded G and H contain inorganic materials which are water soluble and as such are not within the scope of the present invention. These particular Examples are included for comparative purposes.

A series of tablets were prepared at a standard density of 1325 ±10kg/m^3. Tablets 1A to 1H contain 2% by weight of granules A to H respectively, the other 98% being formed from concentrated Persil original non-Biological detergent powder. Tablet 1J was fabricated entirely from the same Persil powder. Tablets 1K and 1L were prepared from the same Persil powder, but incorporated 0.2% and 2% by weight of Aquasorb A500 powder respectively.

Table 3 shows the level of disintegration and the conductivity obtained after 10 minutes immersion in water for these tablets using the experimental protocol described above in Method 1. The conductivity measurements are representative of the degree of dissolution of the soluble ionic constituents of the Persil detergent powder, the higher the conductivity value the greater the degree of tablet dissolution. The measurement of disintegration shows the level of undisintegrated residue retained in the 'cage'. Thus a high value indicates a poorly disintegrating tablet.

TABLE 3

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
1A	3800	5
1B	4100	0
1C	4000	0
1D	3970	0
1E	3700	2
1F	3520	10
1G	1060	84
1H	2840	29
1J	1720	49
1K	1800	48
1L	630	80

The data clearly demonstrate the following:

1) The Tablet 1J, composed entirely of concentrated Persil powder, does not disintegrate or dissolve well; after 10 minutes it is only about 50% disintegrated.
2) Addition of 0.2% Aquasorb A500 (Tablet 1K) has no appreciable effect (this is equivalent to the amount of Aquasorb added via granule incorporation in Tablets 1A to 1D, 1G and 1H).
3) Addition of more Aquasorb in Tablet 1L (equivalent to the total weight addition of granules in Tablets 1A-1H) appears to suppress disintegration and dissolution.
4) Tablets 1A-1D are essentially fully disintegrated/dissolved after 10 minutes.
5) In contrast, when the Aquasorb has been co-granulated with a soluble material (Tablets 1G, 1H), the disintegration and dissolution is far less effective. In the case of silicate, the granules actually appear to retard disintegration relative to Tablet 1J.
6) Even when the level of Aquasorb in the granules is reduced to 7.5% or 5% (Tablets 1E, 1F, respectively) disintegration of the tablets is very well advanced after 10 minutes.

EXAMPLE 2

Table 4 below shows data for Tablets 2E, 2F equivalent to the data in Table 3. These tablets which are similar to the tablets used in Example 1 but contain granules E and F respectively, incorporated into tablets at 4% by weight instead of 2%. The test method for measuring disintegration was Method 1. Again, it is clear that, by increasing the weight of granule in the tablet at these lower levels of Aquasorb inclusion, the excellent disintegrating properties of the granules can be retained without the need to significantly increase the total weight of disintegrating polymer in the tablet (since 4% inclusion of granule F corresponds to 2% of granule D in terms of the total weight of Aquasorb introduced into the granule).

TABLE 4

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
2E	3950	0
2F	3900	0

EXAMPLE 3

Table 5 below shows further data for disintegration/conductivity (using disintegration Method 1) for tablets containing various of the granules, again incorporated into the tablets at 2% by weight. These tablets are identified as 3X, where the X is the letter corresponding to the granule identity in Table 2. In this case, the tablets have been prepared at a higher density (1350 ± 10 kg/m^3). At this higher density, it is possible to discriminate between the performance of the different granules A to D. From this data, it appears that the zeolite-containing granules are preferred over the silica-containing variant, with the granules prepared from Doucil A24 being the best performing disintegrating agents.

TABLE 5

Tablet Code	Conductivity (microSiemen)	Undisintegrated Residue (%)
3A	3060	21
3B	3500	9
3C	3060	13
3D	3860	9

EXAMPLE 4

A number of tablets were prepared using a standard detergent base powder, similar to concentrated Persil powder, but without minor additives, such as perfume. Tablets containing disintegrant granules as shown in Table 6 below were prepared at a nominal density of 1250 kg/m^3 with 5% by weight of granules in the final detergent composition. The disintegrant granules used were all based on the zeolite P, Doucil A24, which consisted of 90% aluminosilicate and 10% water by weight.

TABLE 6

Granule Code	Water-swellable agent in granule (% by weight)
4M	Aquasorb A500 (7.5)
4N	Ac-Di-Sol[1] (5.0)
4O	Ac-Di-Sol[1] (7.5)
4P	Ac-Di-Sol[1] (10.0)
4Q	Ac-Di-Sol[1] (15.0)
4R	Cellulose powder BFT[2] Fines (7.5)
4S	National 78- 1551[3] (7.5)
4T	Arbocel FT40[4] (7.5)

[1]Ac-Di-Sol is a croscarmellulose sodium available from FMC Corporation, Philadelphia USA.

[2]Cellulose powder BFT is a granulated sulphite cellulose available from Vendico Chemical AB, Malmö, Sweden.

[3]National 78-1551 is a pre-gelatinised starch available from National Starch & Chemical, Manchester, UK.

[4]Arbocel FT40 is a fibrous natural cellulose available from J. Rettenmaier & Söhne, Rosenberg, Germany.

The disintegration of the tablets was measured using Method 2, described hereinbefore, and the results are given in Table 7 below.

TABLE 7

Disintegrant code	Undisintegrated residue (%w/w)
4M	40
4N	42
4O	32
4P	36
4Q	34
4R	33
4S	32
4T	43
no disintegrant	87

In general, the amount of residue found using Method 2 is larger than that found using Method 1, but the results still clearly demonstrate that the tablets containing disintegrant granules disintegrate to a much larger extent than tablets containing no granules.

CLAIMS

1. A cleaning composition, the composition including disintegrant in the form of granules comprising a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular disintegrant, the granules being combined with the active ingredients of the cleaning composition in a compacted moulded body.
2. A cleaning composition in which the water-swellable agent comprises, in its anhydrous state, no more than 15% of the combined weight of said inorganic material and said agent of the granular disintegrant.
3. A cleaning composition in which the water-swellable agent comprises, in its anhydrous state, no more than 10% of the combined weight of said inorganic material and said agent of the granular disintegrant.
4. A cleaning composition in which the water-swellable agent comprises, in its anhydrous state, no more than 8% of the combined weight of said inorganic material and said agent of the granular disintegrant.
5. A cleaning composition in which the water-swellable agent comprises, in its anhydrous state, at least 1% of the combined weight of said inorganic material and said agent of the granular disintegrant.
6. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material comprises a silica.
7. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material acts as functional ingredient of the cleaning composition.
8. A cleaning composition as claimed in any one of the preceding claims in which the inorganic material comprises an aluminosilicate.
9. A cleaning composition as claimed in Claim 8 in which the inorganic material comprises a zeolite.
10. A cleaning composition as claimed in Claim 9 in which the inorganic material comprises a zeolite P.
11. A cleaning composition as claimed in any one of Claims 7 to 10 in which the inorganic material comprises from about 10% to about 60% by weight of the cleaning composition.
12. A cleaning composition as claimed in Claim 11 in which the cleaning composition contains at least 1% by weight of the inorganic material embodied in the form of disintegrant granules.
13. A cleaning composition as claimed in any of the preceding claims in which the water-swellable agent has an average primary particle size of no more than 200 μm.

14. A cleaning composition as claimed in any one of the preceding claims in which the water-swellable agent has a water swelling capacity of at least 5 ml/gram.
15. A cleaning composition as claimed in any one of the preceding claims in which the water-swellable agent is selected from the group consisting of cellulose, cross-linked cellulose, carboxymethyl cellulose, sodium carboxymethyl cellulose, cross-linked sodium carboxymethyl cellulose, pre-gelatinised starch, cross-linked starch and cross-linked polyvinyl pyrrolidone.
16. A cleaning composition as claimed in any one of the preceding claims in which the moulded body consists entirely of said cleaning composition.
17. A cleaning composition as claimed in any one of Claims 1 to 15 in which only part of the moulded body is constituted by said cleaning composition, the remainder of the moulded body being constituted by at least one other ingredient for use in detergent and cleaning applications such as fabric washing, dishware washing, stain removal and water softening.
18. A cleaning composition as claimed in any one of the preceding claims in which the amount of water-swellable agent is less than 2% by weight of the cleaning composition.
19. A cleaning composition as claimed in any one of the preceding claims in which the granular disintegrant is present in an amount in the range 1 to 20% by weight of the total cleaning composition.
20. A granular composition suitable for use in a cleaning composition consisting essentially of a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular composition.
21. A process for the preparation of a cleaning composition comprising forming a granular disintegrant composition consisting essentially of a water insoluble inorganic material and a water-swellable agent which, in its anhydrous state, comprises no more than 20% of the combined weight of said inorganic material and said agent of the granular composition and mixing said granular composition with one or more detergent-active compounds and, optionally, with one or more detergency builders.
22. A process as claimed in claim 21 in which the detergent-active compound is a soap, a synthetic non-soap anionic compound or a synthetic non-soap nonionic compound.

INTERNATIONAL SEARCH REPORT

Internat. Application No
PCT/GB 00/02785

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C11D17/00 C11D3/12 C11D3/22 C11D17/06 C11D3/37

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	WO 98 55583 A (UNILEVER) 10 December 1998 (1998-12-10) cited in the application claims 1-15; examples 1B,2	1,2,6-9, 11,15,16
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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
18 October 2000	06/11/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Loiselet-Taisne, S

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INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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